

SYNTHESIS, IDENTIFICATION AND STUDY OF EXPECTED BIOLOGICAL ACTIVITY FOR SOME (SULFIDE, SULFONE) COMPOUNDS

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ABSTRACT

The synthesis of various Sulfide and Sulfone compounds (some of them cyclic and other normal compounds) exhibit a wide range of biological properties. All steps of reactions are followed by TLC papers. All the synthesized compounds have been investigated using different chemical techniques, such as (¹H.NMR–spectra, (C.H.N)-analysis, FT.IR–spectr) ome of them in ¹³C.NMR–spectra, melting points and biological study

KEYWORDS: Sulfone, Sulfide, Biological Study, Heterocycle, Sulfur

INTRODUCTION

Most of the organic compounds containing Sulfur atoms and Sulfone groups in there structures have been used in pharmaceutical application as insecticide like Sulfa drugs ,Sulfonamide ,Sulfadiazine ,Thiazine ,Thiazole.....etc .which exhibited various kind of pharmaceutical activity(1-5) like blood platelet aggregation inhibitors ,antimicrobial ,anti cancer, etc

Production and application of organo sulfide and sulfone compounds are useful in technical and industrial growth like in rubber vulcanization, vitamins, polymers and the other fields (6-8).

Some of studies on the synthesis and biological activities of the Sulfone compounds appeared particularly after the discovery of several broad spectrum anthelmintic compounds (9).

Experiment and Apparatuses

All chemicals used (purity 99.98%), FT.IR-spectra: were recorded on shimadzu 8300, KBr -disc, ¹HNMR-spectra were recorded on varian 300MHz spectrometer using TMS as an internal standard and elemental analysis (C.H.N)-elemental (analyses system GmbH)-measurements and ¹³C.NMR–spectra, were carried out in Canada. The melting points were determined in open capillary tubes by electro thermal 9300LTD, UK. , biological study carried out in Bio – lab in biological department.

Synthesis of Compounds {1- 4}

A mixture of thiosemicarbazide (0.02 mol ,1.82 gm) and diethyl triphthalate (0.01 mol, 2.22 gm) were refluxed for (4hrs) in ethanol abs ,then the precipitate was filtered off and re crystallized from ethanol to yield (82%) of compound {1},which (0.01 mol) reacted with (5ml) of sodium hydroxide (10%) were refluxed for (4hrs) in ethanol abs, then the precipitate was filtered off and re crystallized from ethanol to yield (80%) of compound {2}, which (0.01 mol)reacted with (0.02 mol) of chloro 2- ethyl acetate and (0.01mol) of potassium carbonate were refluxed for (6hrs) in ethanol abs, then the

precipitate was filtered off and re crystallized from ethanol to yield (79%) of compound {3}, which (0.001 mol)reacted with (20ml)of (hydrogen peroxide 60%) in presence of acetic acid for (3hrs), then the precipitate was filtered off and re crystallized from ethanol to yield (81%) of compound {4} .

Synthesis of Compounds {5 -7}

A mixture of sulfobenzoic anhydride (0.01 mol, 1.84gm) and 2-amino -5-mercapto -1,3,4-thiadiazole(0.01 mol ,1.33 gm) were refluxed for (5hrs) in acetone, then the precipitate was filtered off and re crystallized from to yield (80%) of compound {5}, which (0.01 mol)reacted with (0.01 mol) of chloro 2- ethyl acetate were refluxed for (4hrs) in ethanol abs, then the precipitate was filtered off and recrystallized from ethanol to yield (81%) of compound {6}, which (0.01 mol) reacted with (20ml) of (hydrogen peroxide 60%) in presence of acetic acid for (3hrs), then the precipitate was filtered off and recrystallized from ethanol to yield (81%) of compound {7}

Synthesis of Compounds {8 -10}

A mixture of phenylene diamine (0.01 mol ,1.8gm) and (2-mercapto acetic acid) (0.01mol ,0.92gm) and (5ml) of hydrochloric acid were refluxed for (5hrs) in ethanol abs, then the precipitate was filtered off and re crystallized from ethanol to yield (81%) of compound {8}, which (0.02 mol)reacted with(0.01mol ,0.3gm,) of formaldehyde and (5ml) of Sulfuric acid were refluxed for (3hrs) in ethanol abs, then the precipitate was filtered off and recrystallized from ethanol to yield (80%) of compound {9}, which (0.01 mol) reacted with (20 ml) of (hydrogen peroxide 60%) in presence of acetic acid for (3hrs), then the precipitate was filtered off and recrystallized from ethanol to yield (81%) of compound {10}

Synthesis of Compounds {11 -12}

A mixture of (2-amino-5-mercapto-1,3,4-thiadiazol) (0.02 mol ,2.66 gm,) and formaldehyde(0.01 mol ,0.3gm,) and (5ml) of Sulfuric acid were reacted for (3hrs) in ethanol abs, then the precipitate was filtered off and re crystallized from ethanol to yield (82%) of compound {11}, which (0.01 mol)reacted with(0.02 mol ,3.68 gm) of (sulfobenzoic anhydride) were refluxed for (4hrs) in ethanol abs, then the precipitate was filtered off and re crystallized from ethanol to yield (83%) of compound {12}.

Synthesis of Compounds {13 -16}

A mixture of formaldehyde (0.01 mol,0.3 gm,) and (sodium hydroxide 10%) (5ml) and (0.02 mol ,3.08 gm) of(2-mercapto benzoic acid) were rotated for (4hrs) in ethanol abs, then the precipitate was filtered off and re crystallized from ethanol to yield (84%) of compound {13}, which (0.01 mol)reacted with (0.02 mol ,1.82 gm) (thio semicarbazide) and (sodium hydroxide10%) (5ml) were refluxed for (6hrs) in ethanol abs ,then the precipitate was filtered off and re crystallized from ethanol to yield (81%) of compound {14}, which (0.005 mol) reacted with(0.01 mol) of (chloro 2- ethyl acetate) were refluxed for (3hrs) in ethanol abs, then the precipitate was filtered off and re crystallized from ethanol to yield (82%) of compound {15}, which(0.005) reacted with (20ml)of(hydrogen peroxide 60%) in presence of acitic acid for (3hrs), then the precipitate was filtered off and re crystallized from ethanol to yield (78%) of compound {16} .

Synthesis of Compounds {17-19}

A mixture of (2-mercapto benzoic acid) (0.02 mol ,3.08 gm) and (3-chloro propanoyl chloride) (0.01 mol ,1.26 gm) and (0.01 mol ,1.38 gm) of potassium carbonate were refluxed for (6hrs) in ethanol abs, then the precipitate was filtered off and re crystallized from ethanol to yield (80%) of compound {17}, which (0.01 mol) reacted with (thio

semicarbazide) (0.02mol) and (0.01 mol ,1.38 gm) of potassium carbonate were refluxed for (4hrs) in ethanol abs, then the precipitate was filtered off and re crystallized from ethanol to yield (82%) of compound {18}, which (0.01 mol) reacted with (chloro-2- ethyl acetate) (0.02 mol) were refluxed for (4hrs) in ethanol abs, then the precipitate was filtered off and re crystallized from ethanol to yield (81%) of compound {19} .

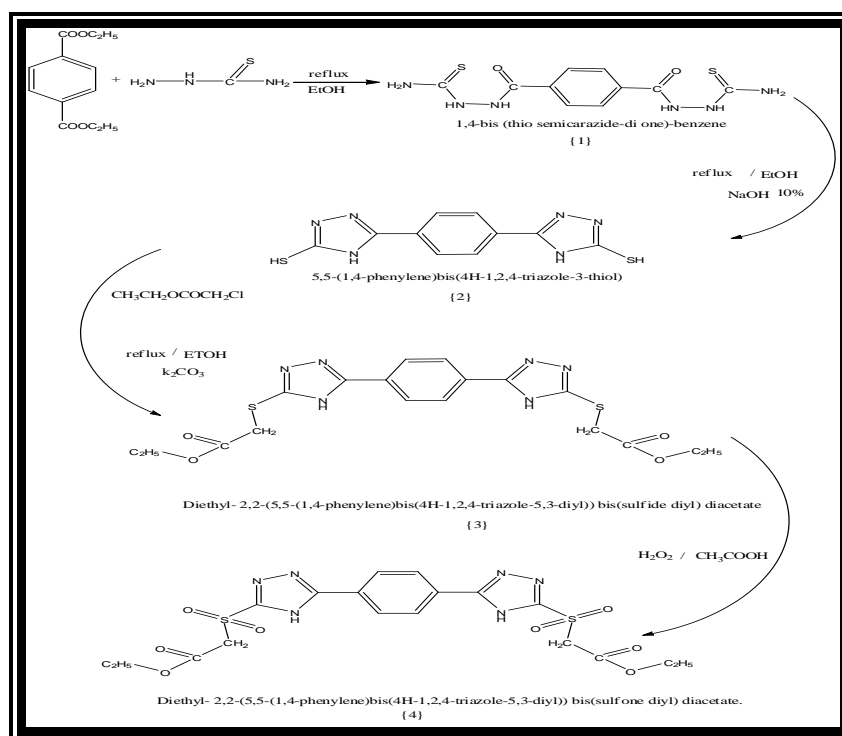


Figure 1: Preparation of Compound {1-4}

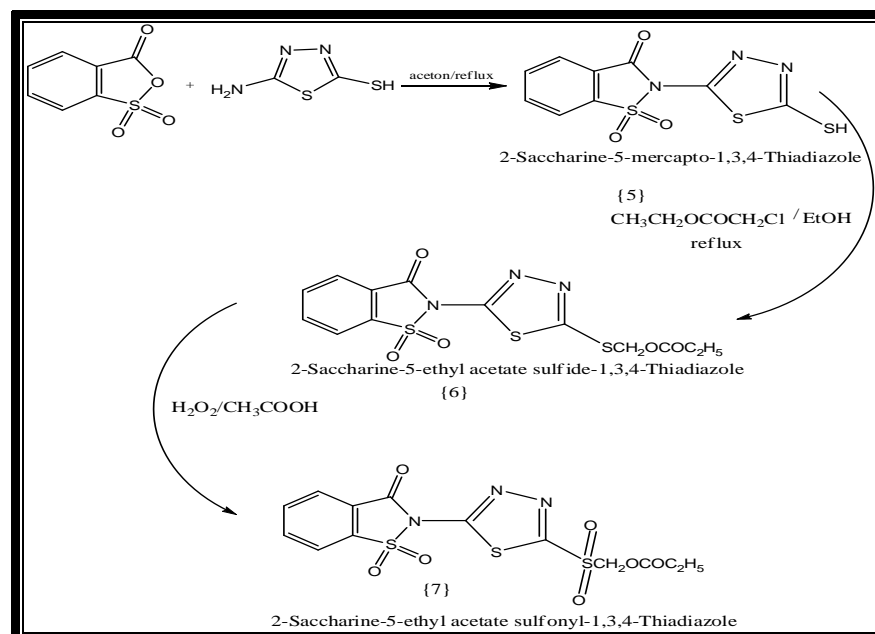


Figure 2: Preparation of Compounds {5 - 7}

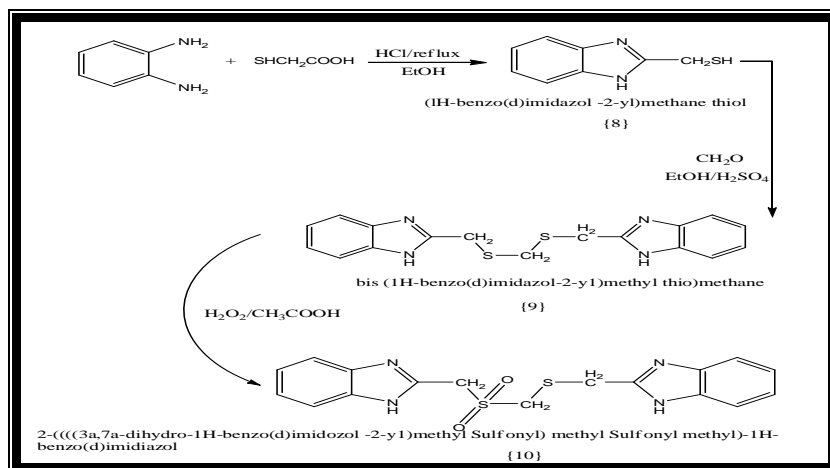


Figure 3: Preparation of Compounds {8 -10}

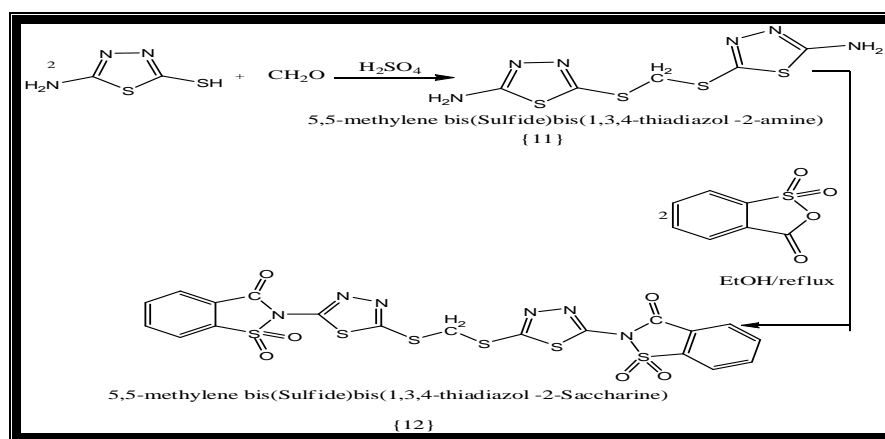


Figure 4: Preparation of Compounds {11-12}

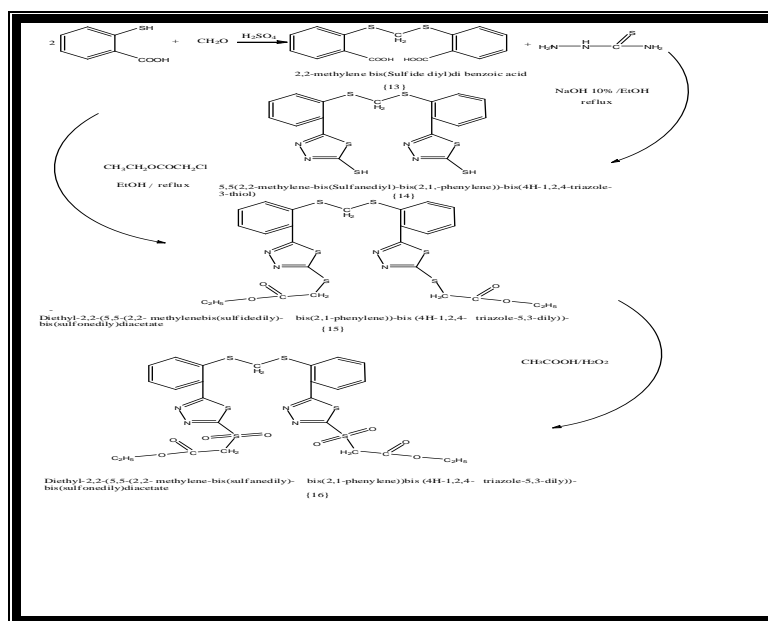


Figure 5: Preparation of Compounds {13 -16}

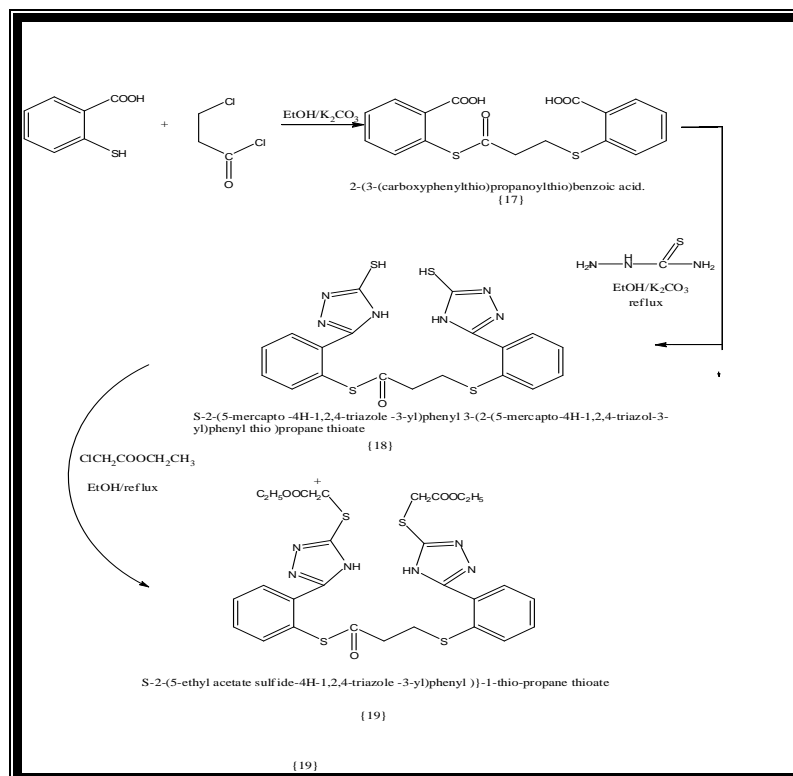


Figure 6: Preparation of Compounds {17 -19}

Biological Activity of Compounds [1-10]

Some of the prepared compounds are tested for the biological activities [1-10], the antibacterial activity (0.2ml) of the synthesized compounds were tested against both Gram-negative and Gram-positive bacteria using their (1×10^2) concentration in DMSO as a solvent. The inhibition zone against the growth of the verified bacteria for the compounds is given in table (1)

Table 1: Effect of Compounds on Inhibition Zone of Bacteria in (Mm.)

Compounds	Inhibition In Mm	
	Staphylococcus Aureus (+)	E-Coli (-)
R₁	10	6
R₂	24	18
R₃	26	18
R₄	28	18
R₅	24	20
R₆	28	16
R₇	30	22
R₈	18	12
R₉	24	16
R₁₀	34	28

RESULTS AND DISCUSSIONS

All synthesized compounds [**R₁ - R₁₉**] were characterized by [FT.IR-spectra, (C.H.N)-analysis, melting points, H.NMR-spectra and some of them by ^{13}C .NMR-spectra].

FT. IR-spectra: showed appearance absorption bands at $(1687) \text{ cm}^{-1}$ ($-\text{CO}-\text{N}$) amide, $(3487, 3389) \text{ cm}^{-1}$ due to amine (NH_2), $(3264) \text{ cm}^{-1}$ to ($\text{NH}-\text{NH}-$), $(1055) \text{ cm}^{-1}$ to ($\text{C}=\text{S}$) in compound [1]. Absorption band at $(1647) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(3416) \text{ cm}^{-1}$ to (NH) of amine group, $(2343) \text{ cm}^{-1}$ to (SH) group in compound [2]. Absorption band at $(1630) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(1200) \text{ cm}^{-1}$ to ($\text{S}-\text{CH}_2$), $(-\text{CO}-\text{O}-)$ to ester group in compound [3]. Absorption band at $(1654) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, absorption band at $(1367) \text{ cm}^{-1}$ due to ($-\text{SO}_2-$) in compound [4]. Absorption band at $(1620) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(771) \text{ cm}^{-1}$ to ($\text{C}-\text{S}$), $(1317) \text{ cm}^{-1}$ to ($-\text{SO}_2$) Sulfone group, $(1698) \text{ cm}^{-1}$ to ($-\text{CO}-\text{N}$) amide, $(2362) \text{ cm}^{-1}$ to ($-\text{SH}$) in compound [5]. Absorption band at $(1616) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(744) \text{ cm}^{-1}$ to ($\text{C}-\text{S}$), $(1384) \text{ cm}^{-1}$ to ($-\text{SO}_2$) Sulfone group, $(1695) \text{ cm}^{-1}$ to ($-\text{CO}-\text{N}$) amide, $(1226) \text{ cm}^{-1}$ to ($\text{S}-\text{CH}_2$), $(1734) \text{ cm}^{-1}$ to ($-\text{CO}-\text{O}-$) ester group in compound [6]. Absorption band at $(1626) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(717) \text{ cm}^{-1}$ to ($\text{C}-\text{S}$), $(1338) \text{ cm}^{-1}$ to ($-\text{SO}_2$) Sulfone group, $(1691) \text{ cm}^{-1}$ to ($-\text{CO}-\text{N}$) amide, $(1726) \text{ cm}^{-1}$ to ($-\text{CO}-\text{O}-$) ester group in compound [7]. Absorption band at $(1608) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(1257) \text{ cm}^{-1}$ to ($\text{S}-\text{CH}_2$), $(3105) \text{ cm}^{-1}$ to ($-\text{NH}$), $(2360) \text{ cm}^{-1}$ to ($-\text{SH}$) in compound [8]. Absorption band at $(1610) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(1220) \text{ cm}^{-1}$ to ($\text{S}-\text{CH}_2$), $(3304) \text{ cm}^{-1}$ to ($-\text{NH}$) in compound [9]. Absorption band at $(1619) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(1377) \text{ cm}^{-1}$ to ($-\text{SO}_2$) Sulfone group, $(3117) \text{ cm}^{-1}$ to ($-\text{NH}$) in compound [10]. Absorption band at $(1627) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(750) \text{ cm}^{-1}$ to ($\text{C}-\text{S}$), $(1267) \text{ cm}^{-1}$ to ($\text{S}-\text{CH}_2$), $(3267, 3184) \text{ cm}^{-1}$ to ($-\text{NH}_2$) group in compound [11]. Absorption band at $(1604) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(796) \text{ cm}^{-1}$ to ($\text{C}-\text{S}$), $(1300) \text{ cm}^{-1}$ to ($-\text{SO}_2$) Sulfone group, $(1685) \text{ cm}^{-1}$ to ($-\text{CO}-\text{N}$) amide, $(1200) \text{ cm}^{-1}$ to ($\text{S}-\text{CH}_2$) group in compound [12]. Absorption band at $(1174) \text{ cm}^{-1}$ due to ($\text{S}-\text{CH}_2$), $(1712) \text{ cm}^{-1}$ to ($-\text{CO}-\text{O}$) carboxylic acid, $(2517-3100) \text{ cm}^{-1}$ to (OH) carboxylic group in compound [13]. Absorption band at $(1633) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(1278) \text{ cm}^{-1}$ to ($\text{S}-\text{CH}_2$), $(3277) \text{ cm}^{-1}$ to ($-\text{NH}$), $(2350) \text{ cm}^{-1}$ to ($-\text{SH}$) group in compound [14]. Absorption band at $(1620) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(1240) \text{ cm}^{-1}$ to ($\text{S}-\text{CH}_2$), $(3201) \text{ cm}^{-1}$ to ($-\text{NH}$), $(1716) \text{ cm}^{-1}$ to ($-\text{CO}-\text{O}$) ester group in compound (15). Absorption band at $(1627) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(1338) \text{ cm}^{-1}$ to ($-\text{SO}_2$) Sulfone group, $(3200) \text{ cm}^{-1}$ to ($-\text{NH}$), $(1726) \text{ cm}^{-1}$ to ($-\text{CO}-\text{O}$) ester group in compound [16]. Absorption band at $(1188) \text{ cm}^{-1}$ due to ($\text{S}-\text{CH}_2$), $(1717) \text{ cm}^{-1}$ to ($-\text{CO}-\text{O}$) group carboxylic acid, $(2546-3150) \text{ cm}^{-1}$ to (OH) carboxylic acid, $(1685) \text{ cm}^{-1}$ to ($-\text{S}-\text{CO}$) group in compound [17]. Absorption band at $(1615) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(1220) \text{ cm}^{-1}$ to ($\text{S}-\text{CH}_2$), $(3304) \text{ cm}^{-1}$ to ($-\text{NH}$), $(2350) \text{ cm}^{-1}$ to ($-\text{SH}$), $(1685) \text{ cm}^{-1}$ to ($-\text{S}-\text{CO}$) group in compound [18]. Absorption band at $(1618) \text{ cm}^{-1}$ due to ($\text{C}=\text{N}$) endo cycle, $(1246) \text{ cm}^{-1}$ to ($\text{S}-\text{CH}_2$), $(3281) \text{ cm}^{-1}$ to ($-\text{NH}$), $(1734) \text{ cm}^{-1}$ to ($-\text{CO}-\text{O}$) ester, $(1696) \text{ cm}^{-1}$ to ($-\text{S}-\text{CO}$) group in compound [19].

Table 2: FT. IR-Data (Cm^{-1}) of Compounds [1-19]

Comp.	(C=N) Endo Cycle	(S-CH ₂) Sulfide	C-S	(S-CO ₂) Sulfone	(CO-N) Amide	Other Groups
{1}					1687	(NH ₂): 3487, 3389 (NH-NH-): 3264 (C=S): 1055
{2}	1647					(NH): 3416 (SH): 2343
{3}	1630	1200				(NH): 3336 (-CO-O-) ester: 1730 (CH) aliph: 2908
{4}	1654			1367		(NH): 3360 (-CO-O-) ester: 1736 (CH) aliph: 2928
{5}	1620		771	1317	1698	(-SH): 2362
{6}	1616	1226	744	1384	1695	(-CO-O-) ester: 1734 (CH) aliph: 2960
{7}	1625		717	1338	1691	(-CO-O-) ester: 1726 (CH) aliph: 2862
{8}	1608	1257				(-SH): 2360

						(-NH):3105
{9}	1610	1220				(-NH):3104
{10}	1619			1377		(-NH):3117 (-CH) aliph:2949
{11}	1627	1267	750			(NH ₂): 3267,3184
{12}	1604	1200	796	1300	1685	
{13}		1174				(-CO-O) carboxylic acid:1712(OH) carboxyl:2517 3100
{14}	1633	1278				(-SH):2350 (-NH):3277
{15}	1620	1240				(-NH):3201 (-CO-O)ester:1726
{16}	1627			1338		(-NH):3200 (-CO-O)ester:1716
{17}		1188				(-CO-O) carboxylic:1717 (OH) carboxylic acid:2546-3150 (S-CO):1685
{18}	1615	1220				(-NH):3304 (S-CO):1672 (-SH):235
{19}	1618	1246				(-NH):3281 (S-CO):1696 (-CO-O)ester:1734

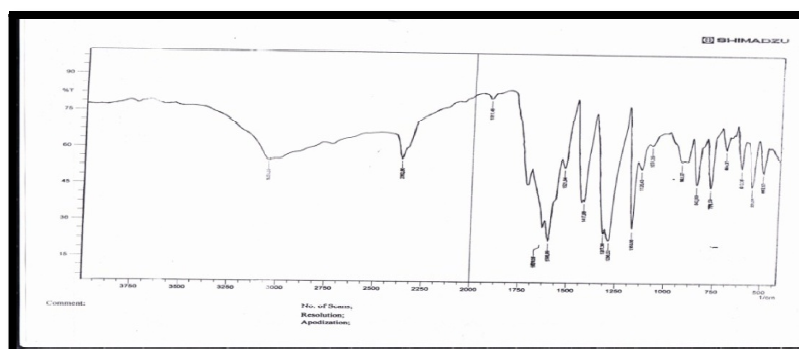


Figure 7: FTIR Spectrum of the Compound {1}

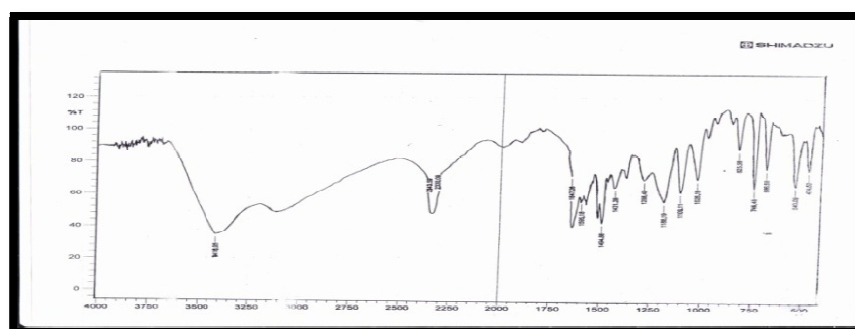


Figure 8: FTIR Spectrum of the Compound {2}

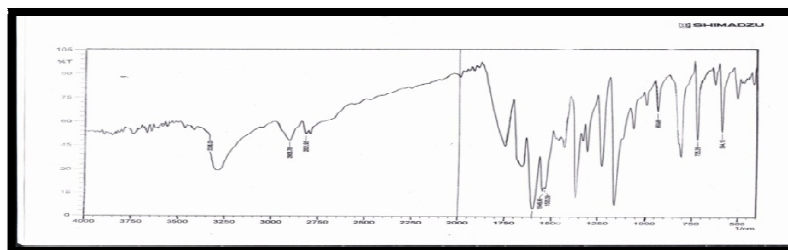


Figure 9: FTIR Spectrum of the Compound {3}

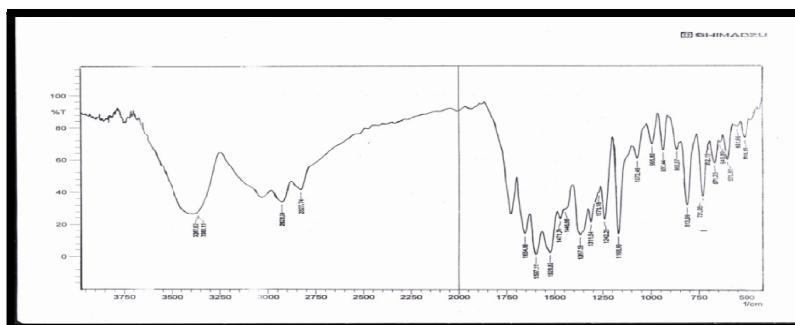


Figure 10: FTIR Spectrum of the Compound {4}

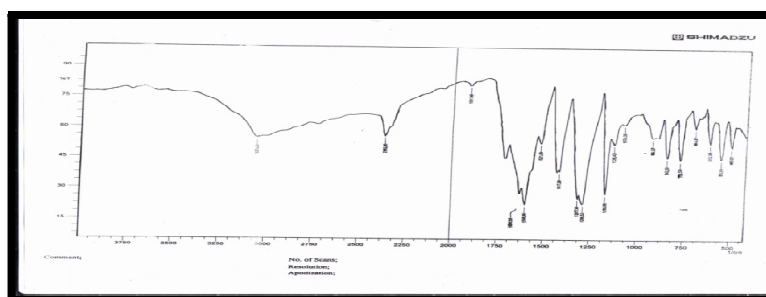


Figure 11: FTIR Spectrum of the Compound {5}

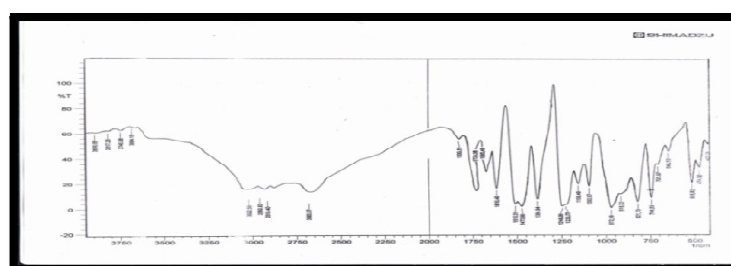


Figure 12: FTIR Spectrum of the compound {6}

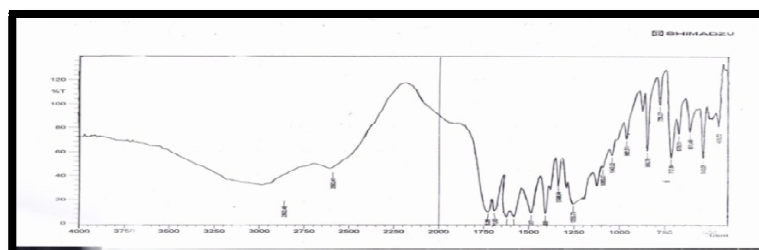


Figure 13: FTIR Spectrum of the Compound {7}

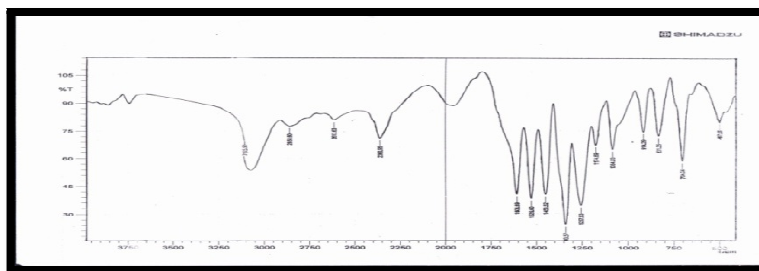


Figure 14: FTIR Spectrum of the Compound {8}

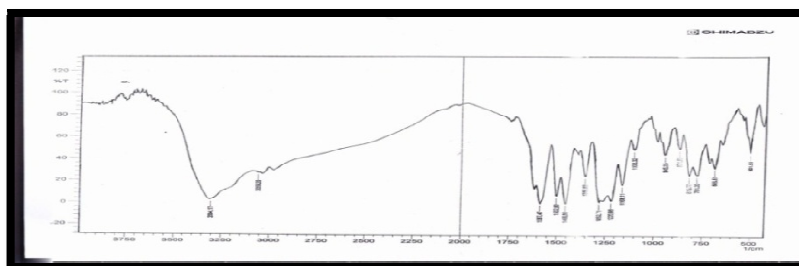


Figure 15: FTIR Spectrum of the Compound {9}

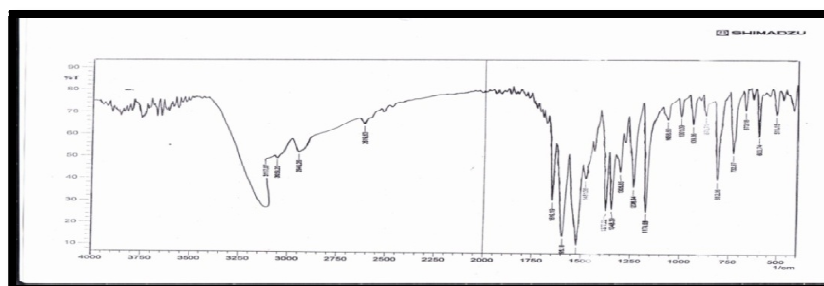


Figure 16: FTIR Spectrum of the Compound {10}

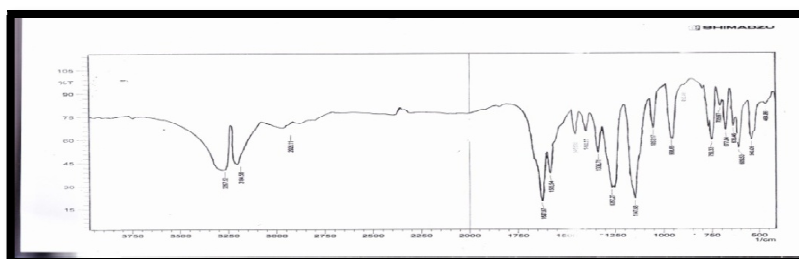


Figure 17: FTIR Spectrum of the Compound {11}

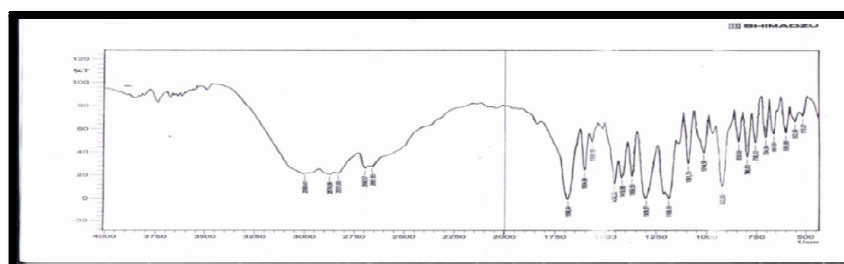


Figure 18: FTIR Spectrum of the Compound {12}

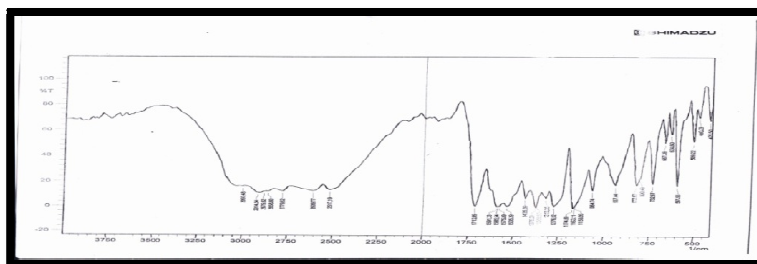


Figure 19: FTIR Spectrum of the Compound {13}

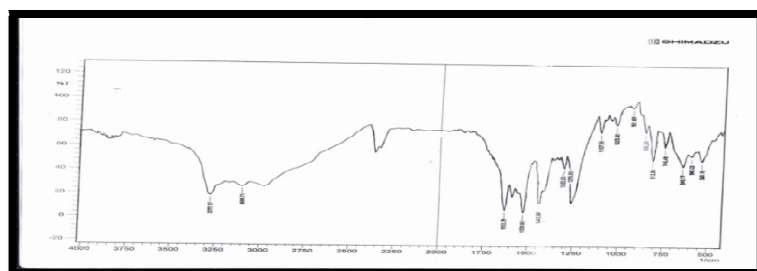


Figure 20: FTIR Spectrum of the Compound {14}

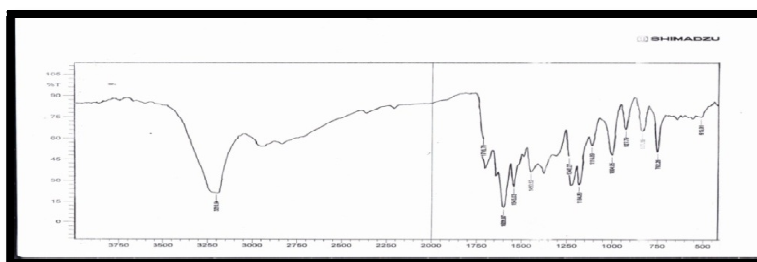


Figure 21: FTIR Spectrum of the Compound {15}

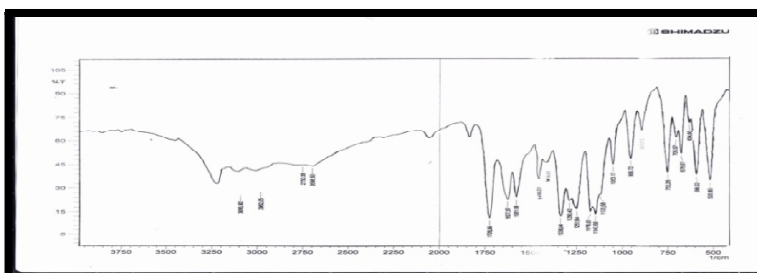


Figure 22: FTIR Spectrum of the Compound {16}

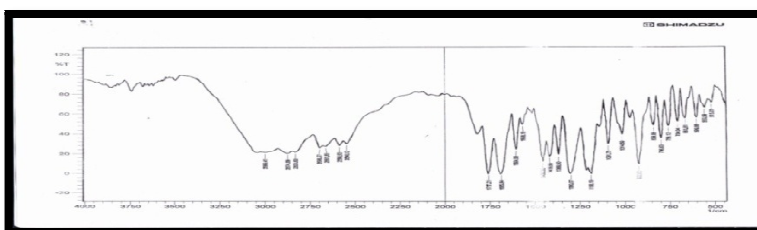


Figure 23: FTIR Spectrum of the Compound {17}

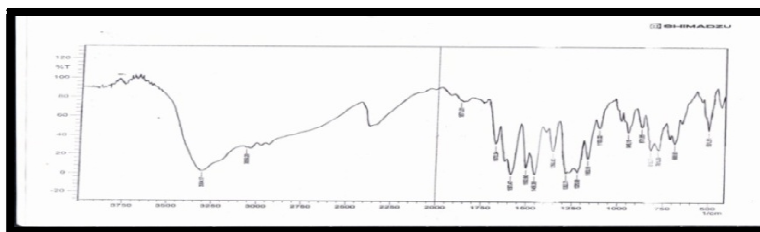


Figure 24: FTIR Spectrum of the Compound {18}

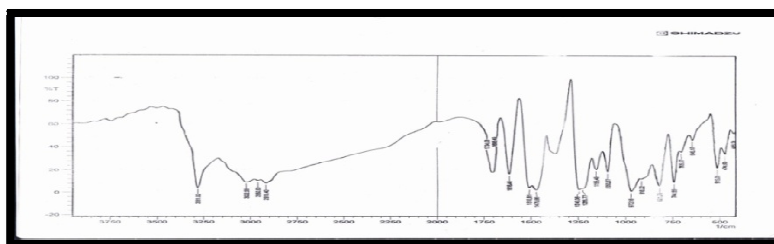


Figure 25: FTIR Spectrum of the Compound {19}

(C.H.N)- Analysis: (C.H.N)- Analysis, from compared the calculated with found data of these compounds, the results were comparable, the data of analysis, MF, names and melting points are in table (3)

Table 3: Physical Properties and (C.H.N)-Analysis of Compounds [1-19]

Comp.	M.F	M.P °c	Found			
			C %	H %	N %	S %
{1}	$C_{10}H_{12}N_6O_2S_2$	148	38.45138.179	3.8683.722	26.90526.713	20.53020.000
{2}	$C_{10}H_8N_6S_2$	169	43.46043.188	2.9152.766	30.41230.204	23.20723.092
{3}	$C_{18}H_{20}N_6O_4S_2$	184	48.20448.054	4.4904.211	18.73818.564	14.29814.153
{4}	$C_8H_{20}N_6O_8S_2$	170	42.18442.079	3.9293.569	16.39816.175	12.51312.308
{5}	$C_9H_5N_3O_3S_3$	154	36.11136.004	1.6811.433	14.03713.889	32.13932.084
{6}	$C_{13}H_{11}N_3O_5S_3$	172	40.51140.204	2.8732.686	10.90210.667	24.95724.717
{7}	$C_{13}H_{11}N_3O_7S_3$	148	37.40537.199	2.6532.304	10.0669.849	23.04422.933
{8}	$C_8H_8N_2S_1$	112	58.51058.274	4.9054.642	17.05816.875	19.52519.334
{9}	$C_{17}H_{16}N_4S_2$	160	59.97459.588	3.7423.482	16.45616.211	18.83618.698
{10}	$C_{17}H_{16}N_4O_4S_2$	158	50.48450.211	3.9833.663	13.85213.554	15.85515.566
{11}	$C_6H_6N_6S_4$	190	24.81524.583	2.0801.951	28.93928.686	44.16544.067
{12}	$C_{20}H_{10}N_6O_6S_6$	192	38.57638.309	1.6171.547	13.49613.277	30.89530.668
{13}	$C_{15}H_{12}O_4S_2$	200	56.23556.104	3.7713.517	/	20.01719.898
{14}	$C_{17}H_{14}N_6S_4$	220	47.42047.199	3.2743.187	19.51819.329	29.78729.538
{15}	$C_{25}H_{26}N_6O_4S_4$	225	49.81749.552	4.3434.178	13.94213.766	21.27821.194
{16}	$C_{25}H_{26}N_6O_{12}S_4$	230	41.09140.849	3.5823.357	11.50011.321	17.55117.319
{17}	$C_{17}H_{14}O_5S_2$	130	56.34156.198	3.8903.644	/	17.69517.465
{18}	$C_{19}H_{16}N_6O_1S_4$	153	48.28548.082	3.4093.277	17.78217.566	27.13727.041
{19}	$C_{27}H_{28}N_6O_5S_4$	161	50.29450.075	4.3724.255	13.03412.954	19.89119.676

The 1H -NMR spectra : showed signals at δ (5.29) for protons of amine group (NH_2) of triazole cycle and signal at δ (12.0) for protons of thiol group (SH) in compound [2]., which converted to signals at δ (4.78) for protons of (S- CH_2 -

) and δ (3.34 --4.23) for protons of ester groups ($\text{C}_2\text{H}_5\text{-COO}$) } in compound [3] ., signals at δ (4.91) for protons of ($\text{SO}_2\text{-CH}_2\text{-}$) and δ (3.0 --4.5) for protons of ester groups ($\text{C}_2\text{H}_5\text{-COO}$) } in compound [4] ., signal at δ (12.0) for proton of thiol group (SH) in compound [5]., signals at δ (4.43) for protons of ($\text{S-CH}_2\text{-}$) and δ (3.0 --4.11) for protons of ester groups ($\text{C}_2\text{H}_5\text{-COO}$) } in compound [6] ., signals at δ (4.48) for protons of ($\text{SO}_2\text{-CH}_2\text{-}$) and δ (3.0 --4.11) for protons of ester groups ($\text{C}_2\text{H}_5\text{-COO}$) } in compound [7] ., at δ (5.61) for protons of amine group (NH_2) and signal at δ (4.35) for protons of ($\text{S-CH}_2\text{-S}$) in compound [11]., signal at δ (4.41) for protons of ($\text{S-CH}_2\text{-S}$) in compound [12]., signal at δ (3.88) for protons of ($\text{S-CH}_2\text{-S}$) and signal at δ (14.91) for proton of (COOH) carboxyl group in compound [13] ., and other signals of functional groups⁽¹⁰⁻¹⁶⁾ in table (4) .

Table 4: ^1H -NMR- Data (δ - Ppm) of Compounds

Comp. No.	^1H -NMR ((Important Peaks))
{2}	5.29 (H , NH) protons of amine triazole ., 12.0 (H , SH) proton of thiol group., (6.81-7.58) for protons of phenyl ring .
{3}	5.59 (H , NH) protons of amine triazole ., 4.78 (H , S-CH ₂), (4.23 -3.34) (H ,C ₂ H ₅ -COO-)protons of ester .,(6.81-7.58) for phenyl ring .
{4}	5.15 (H , NH) protons of amine triazole ., 4.91 (H , SO ₂ -CH ₂), (4.3 -3.20) (H ,C ₂ H ₅ -COO-)protons of ester .,(6.74-7.51) for phenyl ring .
{5}	12.0 (H , SH) proton of thiol group., (6.68-7.56) for phenyl ring .
{6}	4.43 (H , S-CH ₂), (4.11 -3.0) (H ,C ₂ H ₅ -COO-)protons of ester .,(6.87-7.06) for protons of phenyl ring .
{7}	4.48 (H , SO ₂ -CH ₂), (4.11 -3.01) (H ,C ₂ H ₅ -COO-)protons of ester .,(7.04-7.26) for phenyl ring .
{11}	5.61 (H , NH ₂) proton of amine group ., (4.35) (S-CH ₂ -S) .
{12}	4.41 (S-CH ₂ -S).,(7.20-7.44) Phenyl ring.
{13}	14.91 (H , COOH) proton of carboxyl. 3.88 (S-CH ₂ -S).,(7.26-7.82) Phenyl ring.
{14}	5.70 (H , NH) protons of amine triazole ., 12.52(H , SH) proton of thiol group., 4.26 (S-CH ₂ -S) ., (6.81-7.58) for protons of phenyl ring .
{15}	5.96 (H , NH) protons of amine triazole ., (4.88-4.20) (H , S-CH ₂)sulfide groups ., (2.45 -3.0) (H ,C ₂ H ₅ -COO-)protons of ester .,(7.21-7.77) for phenyl ring .

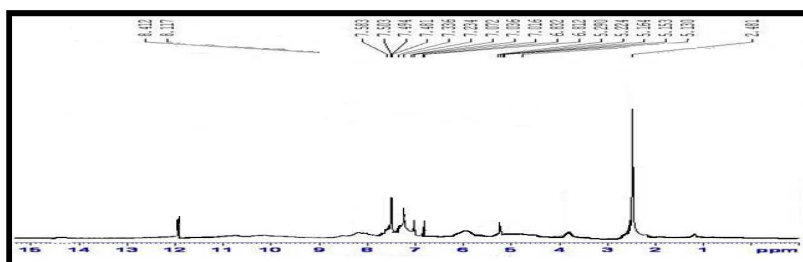


Figure 26: ^1H -NMR of Compound [2]

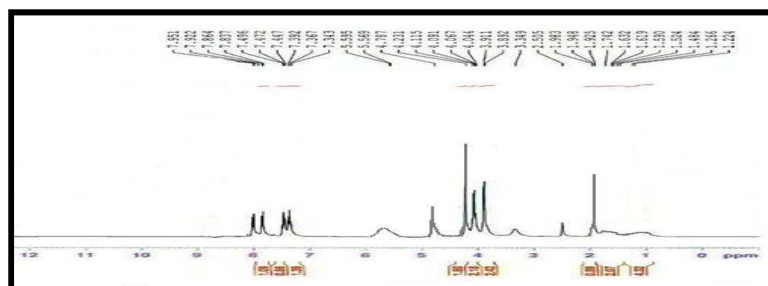
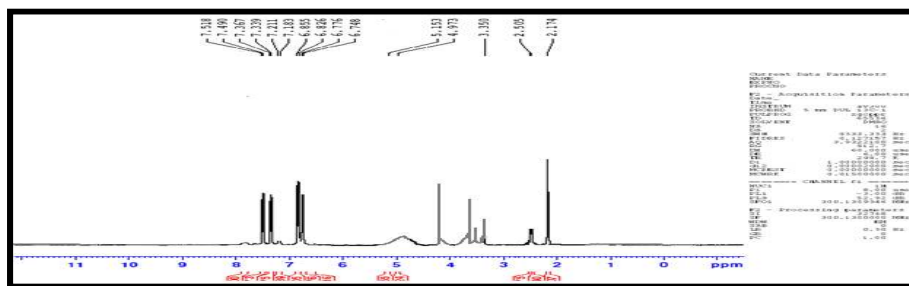
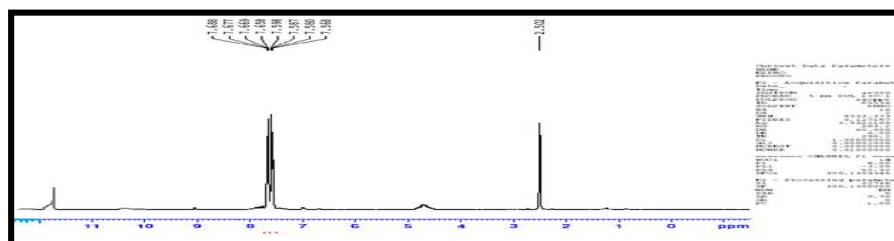
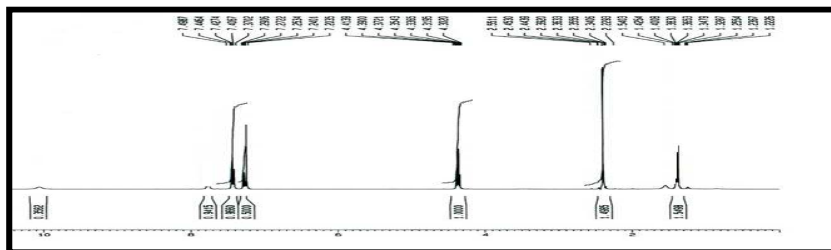
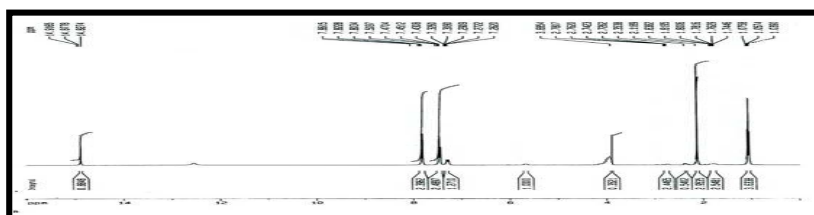
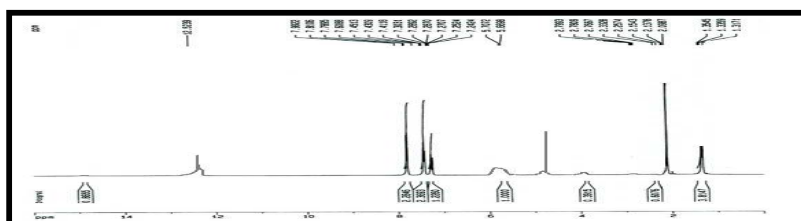
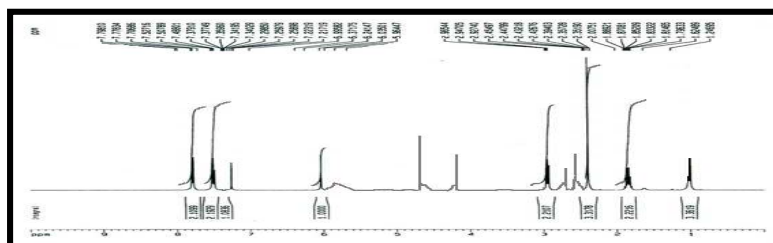


Figure 27: ^1H -NMR of Compound [3]

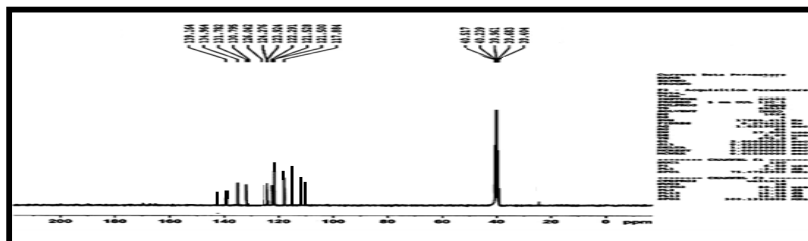
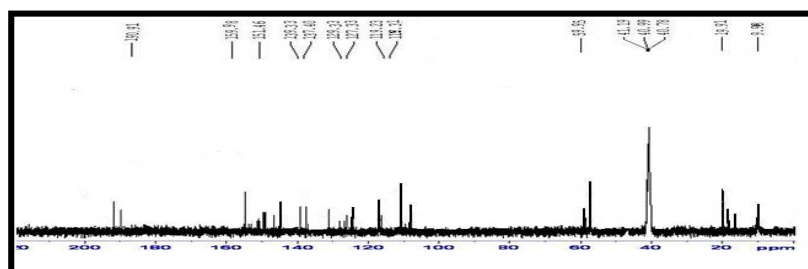
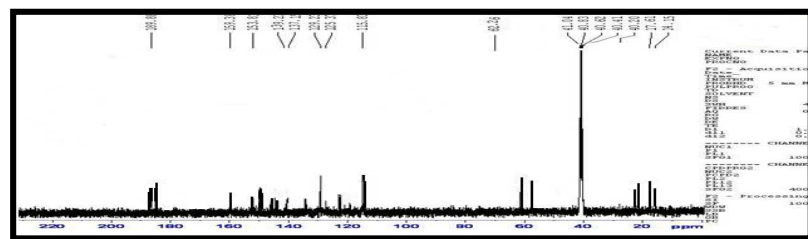
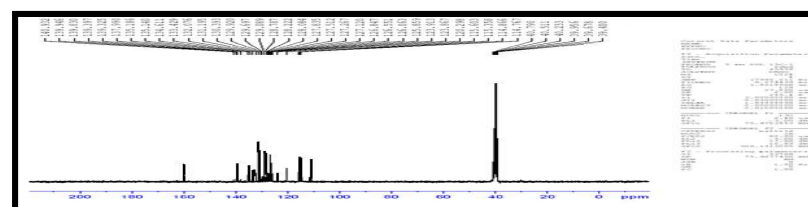
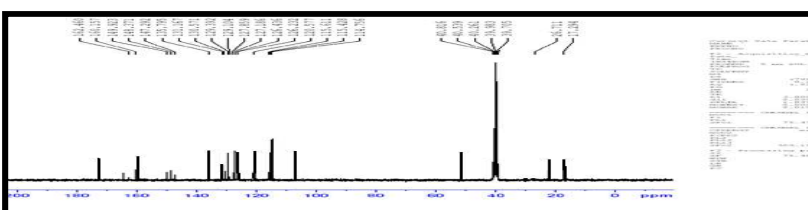
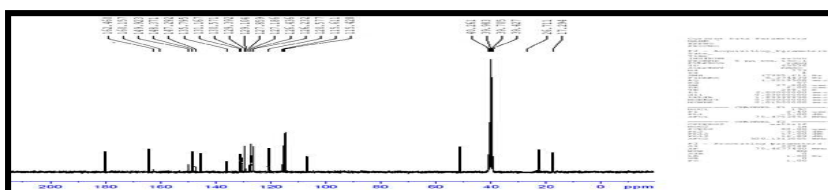
Figure 28: ^1H .NMR of Compound [4]

Figure 33: ^1H -NMR of Compound [12]Figure 34: ^1H -NMR of Compound [13]Figure 35: ^1H -NMR of Compound [14]Figure 36: ^1H -NMR of Compound [15]

The ^{13}C -NMR spectral data of some compounds showed signals indicated to functional groups^(18 -25) in these compounds, table (5).

Table 5: ^{13}C -NMR-Data of Some Compounds

Comp. No.	^{13}C -NMR-Data ((Only Important Peaks))
{ 2 }	(117.8-126) (C , phenyl ring) ,, (130.23-139.69) (C , triazole rings).
{ 3 }	(113-129)(C , phenyl rings) ,,(137-159.6)(C , triazole rings ,, 190 (C ,COO) aster ,, 59.9(C , S-CH ₂) ,, (9.93, 19.19) (C , C ₂ H ₅) ethyl group.
{ 4 }	(115-129)(C , phenyl rings) ,,(137-159.6)(C , triazole rings ,, 189 (C ,COO) aster ,, 62.26(C , SO ₂ -CH ₂) ,, (14.15 , 17.61) (C , C ₂ H ₅) ethyl group.
{ 5 }	(114.8-130)(C ,phenyl ring) ,, (132-140)(C , thiazole rings) ,, 160(C ,CO-N) amide .
{ 6 }	(115-130)(C ,phenyl ring) ,, (131-149)(C , thiazole rings) ,, 162(C ,CO-N) amide ,, 175 (C ,COO) aster ,, 50.0(C , S-CH ₂) ,, (17.29, 26.71) (C ,C ₂ H ₅) ethyl group.
{ 7 }	(114.75 -131)(C ,phenyl ring) ,, (135-149)(C , thiazole rings) ,, 162(C ,CO-N) amide ,, 180.0 (C ,COO) aster ,, 59.0(C ,SO ₂ -CH ₂) ,, (17.29, 30.0) (C ,C ₂ H ₅) ethyl group.
{ 12 }	(108.8 -121.07)(C ,phenyl ring) ,, (136 -147.8)(C , thiazole rings) ,, 168.06 (C ,CO-N) amide ,, (52.88 (C , S-CH ₂ -S)

Figure 37: ^{13}C -NMR of Compound [2]Figure 38: ^{13}C -NMR of Compound [3]Figure 39: ^{13}C -NMR of Compound [4]Figure 40: ^{13}C -NMR of Compound [5]Figure 41: ^{13}C -NMR of Compound [6]Figure 42: ^{13}C -NMR of Compound [7]

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